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The application is published incomplete as filed (Article 93 (2) EPC). The point in the description (Page 6 is missing) or the claim(s) at which the omission obviously occurs has been left blank.

A request for addition of the description (page 6) has been filed pursuant to Rule 88 EPC. A decision on the request will be taken during the proceedings before the Examining Division (Guidelines for Examination in the EPO, A-V, 2.2).

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(54) **A method of forming a sealant having organopolysiloxanes as a main ingredient.**

(57) A method of forming a sealant comprising steps of introducing liquids A and B, which are respectively prepared with specified organopolysiloxanes, into a mixing chamber having a special rotor secured in the inside thereof, mixing the liquids A and B in the mixing chamber by rotating the special rotor, discharging the obtained mixture on the sealing surface of an article, such as an oil pan of a car, and curing it to form the sealant.

The liquids A and B may be cured rapidly and finely to a deep level by mixing them in about equal amounts, and thus they are easily and homogeneously mixed, discharged on the sealing surface, and cured to form the sealant with said mixing chamber, and accordingly, the method is excellent in workability and is applied to on-line systems.

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A METHOD OF FORMING A SEALANT HAVING ORGANOPOLYSILOXANES AS A MAIN INGREDIENT

BACKGROUND OF THE INVENTION

5 1. Field of the Invention:

The present invention relates to a method of forming a sealant applied on a sealing surface of car parts, electric or electronic parts and the like, and more particularly, a method of forming a sealant in which condensing polysiloxanes of two-liquid type, which are possible of curing rapidly and finely to a deep level
10 by mixing the two liquids in about equal amounts, are used as a sealing material, these two liquids may be mixed homogeneously and rapidly with a simple step, and thus, it becomes excellent in workability and is applied to on-line systems.

15 2. Description of the prior Art:

One-liquid type compositions which are cured by condensation are suitable for sealing materials of car parts, electric or electronic parts etc. by the reason that they are very excellent in heat resistant, weather-proofing and electrical properties.

20 Their curing processes are also proposed numerously, and are exemplified as deacetic acid type, deoxime type, dealcohol type, deacetone type and the like.

However, these are all, a type of gradually curing from the surface to the inside by reacting with the moisture in the air, and thus, the curing time becomes long, for example, it requires about 7 to 10 days for curing 10mm, and about 1 month for curing 20mm.

25 Especially, when the FIPG sealant used for the oil pan of a car engine is formed, it is scarcely cured to only the surface thereof since the car engine must be test-driven within about 1 hour from the sealing, and thus, there are some anxiety of troubles, such as oil leakage. Moreover, in case that they are used for adhering and fitting the electric or electronic parts, almost all are used in the on-line systems, and accordingly, said parts should be shipped within several hours after sealing. But, there occurs such
30 inconvenience that they must be left as they are for all day long for curing.

To eliminate the afore-mentioned problems, such a technique that the prior compositions are used as a two-liquid type by dividing them into a main agent and a curing agent, is adopted. But, the amount of the curing agent is very small compared to the main agent, and thus, the mixing thereof is difficult and moreover, the balancing between the main agent and the curing agent is difficult for homogeneous curing.

35 As a result, the slight variation of the amount ratio makes the usable time very short, and thus, they are gelled during the mixing, or they are not cured, or they are cured from the surface just like a one-liquid type, and accordingly, it is scarcely placed into service.

40 SUMMARY OF THE INVENTION

It is an object of the present invention to provide a method of forming a sealant having organopolysiloxanes as a main ingredient wherein the disadvantages found in the prior are overcome.

45 It is another object of the present invention to provide such a method wherein condensing polysiloxanes of two-liquid type, which are possible of curing rapidly and finely to a deep level by mixing the two liquids in about equal amounts, are used as a sealing material.

It is furthermore another object of the present invention to provide such a method wherein said two liquids may be mixed homogeneously and rapidly with a simple step.

50 It is moreover another object of the present invention to provide such a method wherein it is excellent in workability and is applied to on-line systems.

According to the present invention, the above objects are accomplished by providing a method of forming a sealant having organopolysiloxanes as a main ingredient which comprises steps of separately introducing liquids A and B into a mixing chamber in about equal amounts, said liquid A being a mixture of organopolysiloxanes having a radical represented by a general formula of $\text{-SiR}_n\text{X}_{3-n}$ (in the formula, X is a hydrolyzable radical, R is a monohydrocarbon radical, n is 1 or 0) at both terminals of a molecular chain,

and a condensing catalyst, said liquid B being organopolysiloxanes having at least two silanol radicals in one molecule, and said mixing chamber having a rotor secured in the inside thereof and a nozzle formed on the bottom thereof, said rotor being cut on the surface thereof to form grooves, mixing the both liquids in the mixing chamber by rotating the rotor, discharging the obtained mixture on the sealing surface of an article to be coated through said nozzle, and curing it to form a sealant.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 shows a sectional view of one embodiment of the apparatus for practising the method of the present invention, and

Fig. 2 shows another embodiment of the rotor in the apparatus of Fig. 1.

DETAILED DESCRIPTION

The present invention is illustrated in detail with the attached drawings as follows. Fig. 1 shows a sectional view of one embodiment of the apparatus for practising the method of the present invention. The reference numeral 1 shows a mixing chamber. In the inside 1a thereof, a rotor 2 is secured, and on the bottom 3 thereof, a nozzle 4 is formed. Moreover, tanks 7 and 8 are installed thereon to be connected with the inside 1a of the mixing chamber 1 through pipes 5 and 6, and pumps 9 and 10 are also installed on the desired positions of the pipes 5 and 6.

The rotor 2 is of a cylindrical shape tapered at the end 2a as indicated in Fig. 1, having a spindle 11 in a central axis, and is rotatably hung in the inside 1a of the mixing chamber 1 by piercing the top 12 of the mixing chamber 1 with an upper part of the spindle 11.

On the surface of the rotor 2, grooves 13 are cut. The grooves 13 may be in various shapes, such as spiral grooves 13 as mentioned in Fig. 1, or combined grooves of longitudinal grooves 14 cut in plural at a desired distance and spiral grooves 13 as indicated in Fig. 2.

Additionally, the reference numeral 15 is a tank in which washing solvents, such as toluene, trichlene, etc. are contained, and it is connected to the inside 1a of the mixing chamber 1 through pipe 16. On the pipe 16, pump 17 is installed.

In the afore-mentioned apparatus, the sealing material of the condensing polysiloxanes of two-liquid type which are composed of liquids A and B are at first contained separately in the tanks 7 and 8, respectively. Said materials may be cured rapidly and finely to a deep level by mixing the liquids A and B in about equal amounts. The liquids A and B are respectively designated as follows.

[Liquid A]

A mixture of organopolysiloxanes having at both terminals of a molecular chain, a radical represented by a general formula of $-\text{SiR}_n\text{X}_{3-n}$ (in the formula, X is a hydrolyzable radical, R is a monohydrocarbon radical, n is 1 or 0) and a condensing catalyst.

The R radicals of the organopolysiloxanes having at both terminals of the molecular chain, a radical represented by the general formula of $\text{SiR}_n\text{X}_{3-n}$ are monohydrocarbon radicals, and may be exemplarily designated as methyl radical, ethyl radical, propyl radical, vinyl radical, phenyl radical, trifluoropropyl radical etc.. Moreover, the hydrolyzable radicals shown as X are indicated as acyloxy radicals, such as acetoxy radical, octanoyloxy radical, benzoyloxy radical, etc., ketoxime radicals, such as dimethyl ketoxime radical, methyl ethyl ketoxime radical, diethyl ketoxime radical, etc., alkoxy radicals, such as methoxy radical, ethoxy radical, propoxy radical, etc., alkenyloxy radicals, such as isopropenyloxy radical, 1-ethyl-2-methylvinylloxy radical, etc., amino radicals, such as dimethylamino radical, diethylamino radical, butylamino radical, cyclohexylamino radical, etc., aminoxy radicals, such as dimethylaminoxy radical, diethylaminoxy radical, etc., amido radicals, such as N-methylacetamido radical, N-ethylacetamido radical, N-methylbenzamido radical, etc..

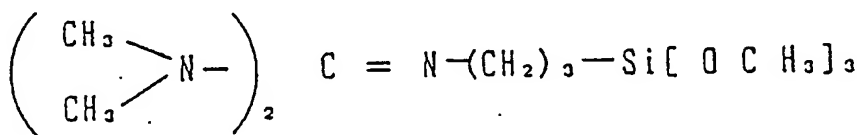
Moreover, the terminal reactive organopolysiloxanes of said ingredient are easily obtained by mixing the corresponding organopolysiloxanes containing terminal silanol radicals with silanes represented by a general formula of $\text{R}_n\text{SiX}_{4-n}$, and heating the same. Said organopolysiloxanes containing terminal silanol radicals are conventionally used as a main raw material of silicone rubber, and are generally closed with hydroxyl groups at both terminals of the molecular chain. There is no limitation to the organic radicals combined with silicon atoms in said organopolysiloxanes, and they are exemplarily shown as alkyl radicals,

such as methyl radical, ethyl radical, propyl radical, butyl radical, etc., cycloalkyl radicals, such as cyclopentyl radical, cyclohexyl radical, etc., alkenyl radicals, such as vinyl radical, allyl radical etc., aryl radicals, such as phenyl radical, tolyl radical, naphthyl radical, etc., aralkyl radicals, such as 2-phenylethyl radical, etc., alkoxy radicals, such as methoxy radical, ethoxy radical, propoxy radical, etc., or their radicals in which a part or all hydrogen atoms are substituted by halogen atoms, such as chlorine atom, or amino radicals, and the like. Said organopolysiloxanes are in the polymerization degree having a viscosity of 100 ~1, 000,000 cS, preferably 1, 000~50, 000 cS at 25°C. The reason resides in that when said diorganopolysiloxanes are below 100 cS (25°C) in viscosity, it becomes difficult to obtain a cured material excellent in physical strength, and to the contrary, when they are above 1, 000, 000 cS (25°C) in viscosity, the viscosity of the composition containing them becomes too high, and as a result, the workability becomes worse when the composition is used in practice.

Moreover, the condensing catalysts (curing catalysts) may be well known catalysts used in this kind of compositions, and are indicated as metallic salts of carboxylic acids, such as lead-2-ethyloctoate, dibutyl tin dioctoate, dibutyl tin diacetate, dibutyl tin dilaurate, butyl tin-2-ethyl hexoate, iron-2-ethyl hexoate, cobalt-2-ethyl hexoate, manganese-2-ethyl hexoate, zinc-2-ethyl hexoate, primary tin caprylate, tin naphthenate, tin oleate, tin butylate, titan naphthenate, zinc naphthenate, cobalt naphthenate, zinc stearate, etc., organic titanate esters, n-aminotitanate, tetra(isopropenyloxy)titanate, etc., organic titan compounds, such as, organosiloxytitan, β -carbonyltitan, etc., aminoalkyl-substituted alkoxysilanes, such as alkoxyaluminum compounds, 3-aminopropyltriethoxysilane, N-(trimethoxy silylpropyl)ethylenediamine, etc., amine compounds and their salts, such as, hexylamine, dodecylamine phosphate, etc., tetraammonium salts, such as, benzyltriethylammonium acetate, etc., lower fatty acid salts of alkali metals, such as, potassium acetate, sodium acetate, lithium oxalate, etc. dialkylhydroxylamines, such as, dimethylhydroxylamine, diethylhydroxylamine, etc., guanidine compounds and guanidyl containing silanes or siloxanes, such as the following formulas, etc..

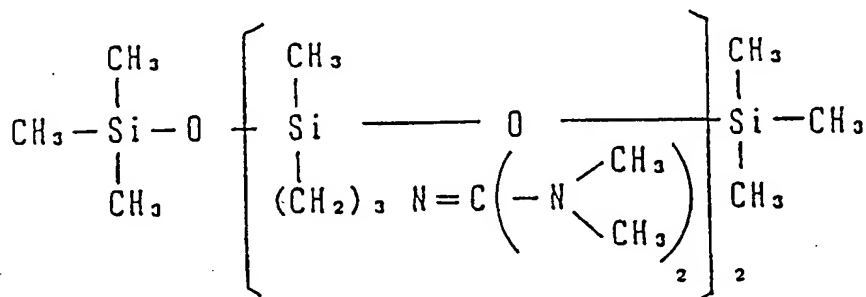
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When they are placed into service, they are not limited in one kind thereof, and they may be used as a mixture of two or more kinds thereof.

The amount of said condensing catalysts to be used is in the range of 0.01~5 parts by weight, preferably 0.1 ~3 part by weight to 100 parts by weight of said organopolysiloxanes. When the amount is too small, it takes long time for curing, and in thick cases, it becomes difficult to homogeneously cure to a deep level, and to the contrary, when the amount is too large, the curing time becomes too short, and thus, there occur many troubles in workability, and moreover, the obtained cured material becomes worse in heat resistant property or weatherproofing property.

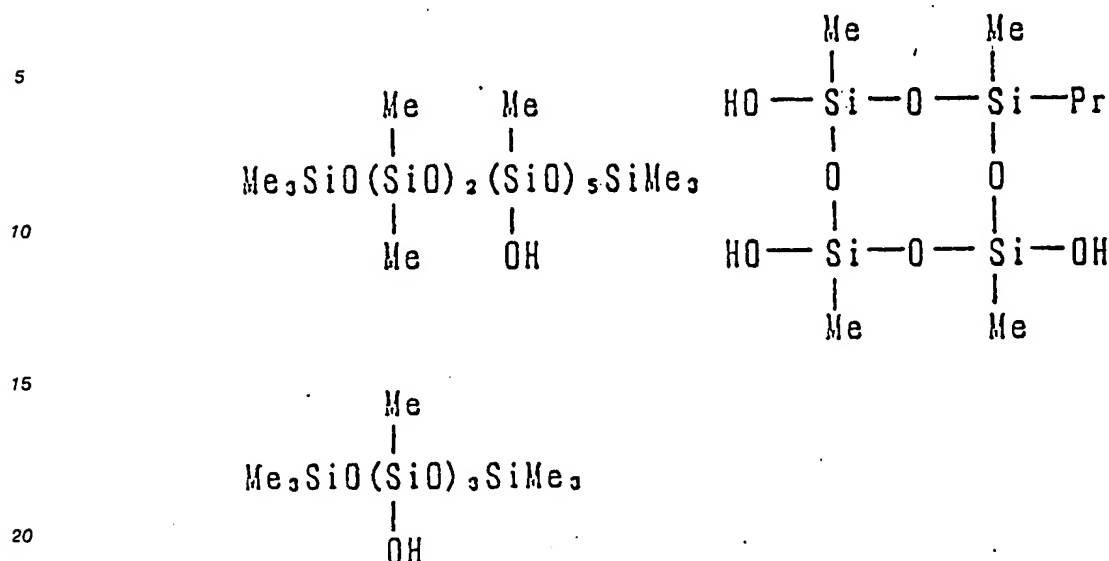
[liquid B]

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Organopolysiloxanes having at least two silanol radicals in one molecule.

The silanol-containing organopolysiloxanes as the liquid B comprise the organopolysiloxanes containing terminal silanol radicals as a raw material of reactive organopolysiloxanes in the liquid A, and moreover,

they may contain more than two silanols in one molecule. They are exemplarily shown as follows.



In said examples, the compounds below 100CS in viscosity may be also used.

The liquid B may be used in the amounts of 3~500 parts by weight, preferably 50~150 parts by weight to 100 parts by weight of the liquid A. then the amount is below 3 parts by weight, it can not give enough amounts of silanols to the reactive radicals of liquid A, and thus, the physical characteristics after curing become worse. Moreover, when the amount is above 500, the silanols remain unreacted, and thus, the heat resistant property becomes extremely worse. In the present invention, the blending rate of said liquid A/liquid B is preferably 1:1 to make easy to blend

In addition, several kinds of fillers may be added in said liquid A or B of the present invention in a suitable amount. The examples of the fillers are metallic oxides, such as, fine silica powder, silica areogel, precipitated silica, diatomaceous earth, iron oxide, zinc oxide, titan oxide, etc., or their metallic oxides treated with silanes on the surface thereof, metallic carbonates, such as, calcium carbonate, magnesium carbonate, zinc carbonate, etc., powders of synthetic resins, such as asbestos, glass wool, carbon black, fine mica powder, melted silica powder, polystyrene, polyvinyl chloride, polypropylene, and the like.

Moreover, into the liquid A or B, thixotropic agents, pigments, dyestuffs, age resisters, antioxidants, antistatic agents, fire retardants, such as antimony oxide, paraffin chloride, etc., agents for improving thermal conductivity, such as, boron nitride, aluminium oxide, etc., rust inhibitors, and the like may be optionally added if necessary, and moreover, various kinds of conventional additives may be mixed therewith, and the examples of said additives are agents for giving adhesive, the so-called carbon functional silanes having amino radical, epoxy radical, thiol radical etc., metallic salts of carboxylic acids, metal alcoholates and the like. Furthermore, they may be diluted with hydrocarbon solvents, such as toluene, petroleum ether, etc., to be easily placed into service.

And then, the liquids A and B are respectively and separately introduced into the inside 1a of the mixing chamber 1 from the tanks 7 and 8 in about equal amounts by the operations of the pumps 9 and 10 through the pipes 5 and 6. At this time, spindle 11 is rotated at 100~2000rpm, preferably 500~1500rpm to rotate rotor 2, and liquids A and B are mixed in the mixing chamber 1. The obtained mixture is descended along with the spiral grooves 13, discharged to make a shape of a sealant on the sealing surface 18a of an article 18 to be coated (for example, an oil pan of a car shown in Fig. 1) from the tip 4a of a nozzle 4, and cured to form a sealant 19. The curing time of said sealant is 10 minutes to start the curing, and 2 hours to be wholly cured to a deep level, at the normal temperature, respectively.

In the above-described present invention, the measurement of liquids A and B in equal amounts is easily accomplished, for example, by constantly operating the pumps 9 and 10 as shown in Fig. 1 since the condensing organopolysiloxanes of two-liquid type used as the sealing material may be cured rapidly and finely to a deep level by mixing the two liquids in about equal amounts. The liquids A and B introduced into the inside 1a of the mixing chamber 1 are homogeneously and rapidly mixed by the grooves 13 formed on the surface of the rotor 2 with the easy step of rotating the rotor 2, and thus, such a trouble that they are cured during the mixing is eliminated even though the curing time of the sealing material becomes short.

Moreover, the mixture never remains in the mixing chamber 1 since it resides only in the grooves.

Accordingly, the present invention is excellent in workability and is applied to on-line systems, and the discharged mixture is homogeneously cured within a short time to a deep level. The present invention will now be described in more detail by way of Example.

5

EXAMPLE

10 Into 100 parts by weight of dimethylpolysiloxane which is closed with silanols at the terminals of a molecular chain, having a viscosity of 20,000CS/25°C, 5 parts of methyltributanooximesilane was added, and heated at 80°C for about 3 hours to synthesize polysiloxane closed with methylbutanooximesilane at the terminals. To 90 parts by weight of said methylbutanooximesilane, 10 parts by weight of a filler (fumed silica) R-972 (manufactured by Nippon Aerogil Co.) and 0.3 parts of dibutyltin dilaurate were added, and mixed homogeneously under N₂ gas current to obtain liquid A.

15 Then, 10 parts by weight of said R-972 were mixed with 90 parts by weight of dimethylpolysiloxane closed with silanols at the terminals of a molecular chain, having a viscosity of 20,000cs/25°C to obtain liquid B.

The liquids A and B thus prepared were respectively and separately charged into the tanks 7 and 8 of the apparatus of Fig. 1, and at the same time, the spindle 11 of the rotor 2 was rotated at a speed of about 200 rpm. Then, the liquids A and B were separately introduced into the inside 1a of the mixing chamber 1 in equal amounts by operating the pumps 9 and 10 with the same power, and were mixed. The mixing was carried out rapidly and homogeneously. The obtained mixture was descended downward through the grooves 13, discharged on the sealing surface 18a of the oil pan 18 of a car through the tip 4a of the nozzle 4, and cured to a deep level after 40 minutes to form the sealant 19. After the finish, washing liquid (such as toluene) was introduced into the mixing chamber 1 by operating the pump 17 through the pipe 16 from the tank 15 to wash the inside of the mixing chamber 1, the rotor 2, the grooves 13 and the like.

25 The obtained sealant 19 was measured for the properties thereof in accordance with JIS-K-6301, and as a result, the hardness was JIS-A25, the tensile strength was 18kgf/cm², and the elongation percentage was 420%.

30 According to the present invention, as mentioned above, the condensing polysiloxanes of two-liquid type used as a sealing material, which are rapidly and finely cured to a deep level by mixing the two liquids in about equal amounts, and the two liquids are homogeneously and rapidly mixed with a simple step, and then, the mixture is discharged on the sealing surface of the article to be coated, and accordingly, the present invention is excellent in workability, applied to the on-line systems, and thus, it is very available for the industry.

35

Claims

- 40 (1) A method of forming a sealant having organopolysiloxanes as a main ingredient which comprises the steps of:
introducing liquids A and B into a mixing chamber separately in about equal amounts, said liquid A being, a mixture of organopolysiloxanes having a radical represented by a general formula of $-\text{SiR}_n\text{X}_{3-n}$ (in the formula, X is a hydrolyzable radical, R is a monohydrocarbon radical, n is 1 or 0) at both terminals of a molecular chain, and a condensing catalyst, said liquid B being organopolysiloxanes having at least two silanol radicals in one molecule, said mixing chamber having a rotor secured in the inside thereof and a nozzle formed on the bottom thereof, said rotor being cut on the surface thereof to form grooves,
45 mixing the both liquids in the mixing chamber by rotating the rotor,
discharging the obtained mixture on the sealing surface of an article to be coated through said nozzle, and
50 curing it to form a sealant.
(2) The method of forming a sealant according to claim 1, wherein said rotor is cut on the surface thereof to form spiral grooves.
(3) The method of forming a sealant according to claim 1, wherein said rotor is cut on the surface thereof to form combined grooves of longitudinal plural grooves and spiral grooves.
55 (4) The method of forming a sealant according to claim 1, wherein the organopolysiloxanes in liquid A are obtained by mixing the corresponding organopolysiloxanes containing terminal silanol radicals with silanes represented by a general formula of $\text{R}_n\text{SiX}_{4-n}$.
(5) The method of forming a sealant according to claim 4, wherein said organopolysiloxanes are in the

polymerization degree having a viscosity of 100~1,000,000 cP at 25°C.

(6) The method of forming a sealant according to claim 1, wherein said condensing catalysts are selected from the group consisting of metallic salts of carboxylic acids, organic titanate esters, organic titan compounds, aminoalkyl-substituted alkoxysilanes, amine compounds and their salts, tetraammonium salts, lower fatty acid salts of alkali metals, dialkylhydroxylamines, guanidine compounds and guanidyl containing silanes or siloxanes.

(7) The method of forming a sealant according to claim 6, wherein the amount of said condensing catalysts to be used is in the range of 0.01~5 parts by weight to 100 parts by weight of said organopolysiloxanes.

(8) The method of forming a sealant according to claim 1, wherein said organopolysiloxanes in liquid B are organopolysiloxanes containing terminal silanols.

(9) The method of forming a sealant according to claim 8, wherein said organopolysiloxanes contain more than two silanols.

(10) The method of forming a sealant according to claim 1, wherein the blending ratio of said liquid A/liquid B is 100:3 ~500 parts by weight, and preferably is 1:1 parts by weight.

FIG. 1

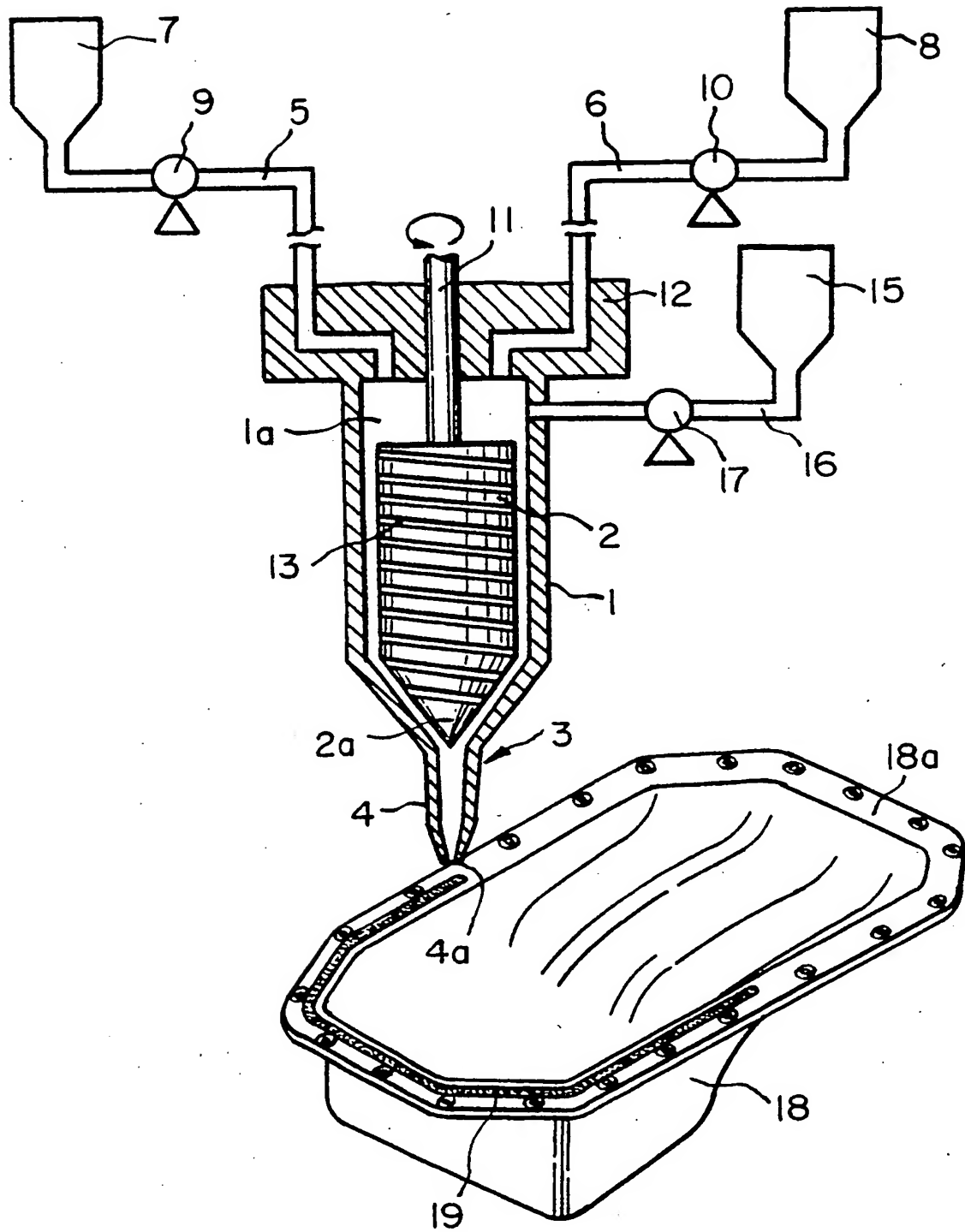
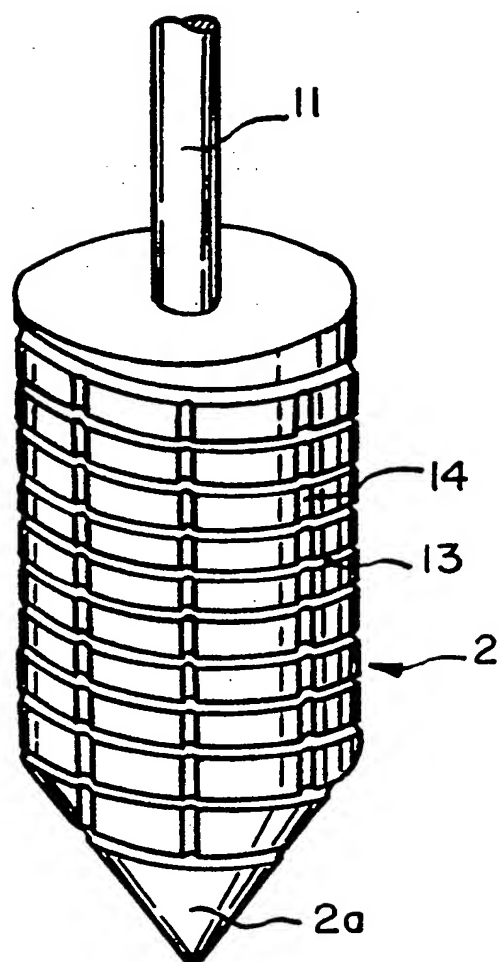


FIG. 2



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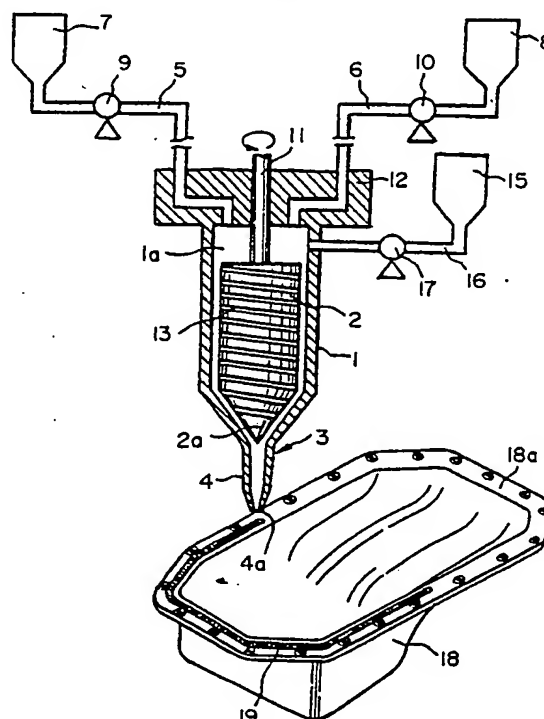
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(54) A method of forming a sealant having organopolysiloxanes as a main ingredient.

(57) A method of forming a sealant comprising steps of introducing liquids A and B, which are respectively prepared with specified organopolysiloxanes, into a mixing chamber having a special rotor secured in the inside thereof, mixing the liquids A and B in the mixing chamber by rotating the special rotor, discharging the obtained mixture on the sealing surface of an article, such as an oil pan of a car, and curing it to form the sealant.

The liquids A and B may be cured rapidly and finely to a deep level by mixing them in about equal amounts, and thus they are easily and homogeneously mixed, discharged on the sealing surface, and cured to form the sealant with said mixing chamber, and accordingly, the method is excellent in workability and is applied to on-line systems.

FIG. 1



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EUROPEAN SEARCH REPORT

Application Number

EP 90 11 3348

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Y	DE-A-2 018 071 (MIDLAND SILICONES) * Page 2; claims *	1,4-10	C 08 L 83/04
Y	QUESTEL TELESYSTEMS (WPI), Derwent accession no. 76-91020X [49], Derwent Publications Ltd, London, GB; & FR-A-2 299 077 (ETUD. REAL CONS. INST.) * Abstract *	1-3	
Y	GB-A-1 020 502 (MIDLAND SILICONES) * Page 2, line 84 - page 3, line 25; claim 1 *	1,4-10	
A,P	EP-A-0 369 259 (ARA-WERK) * Claim 1 *		
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C 08 L C 08 J
The present search report has been drawn up for all claims			
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